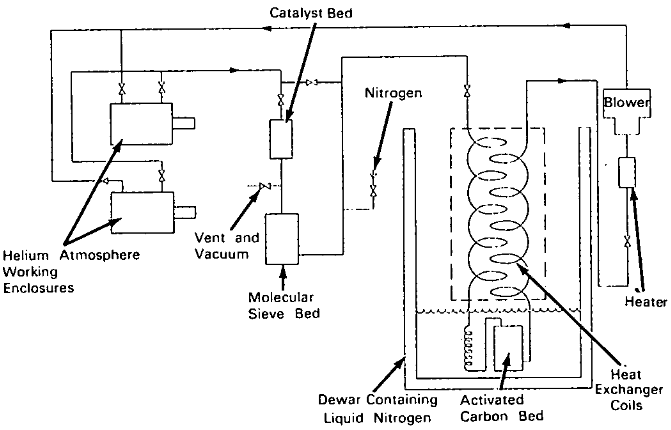
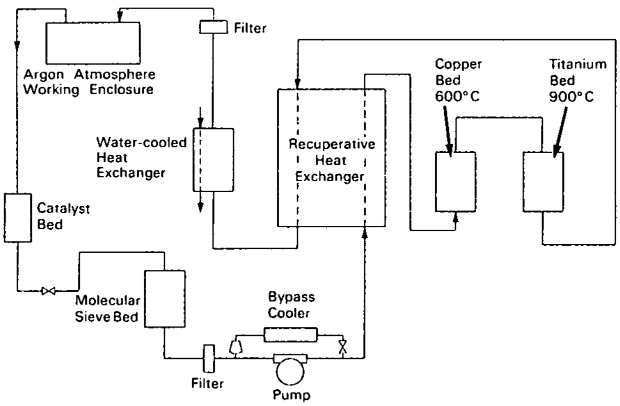
AEC-NASA TECH BRIEF

Two Systems Developed for Purifying Inert Atmospheres





The problem:

To maintain a pure inert gas atmosphere when handling reactive materials. The principal contaminants of such a system are oxygen, nitrogen, and water vapor, and less frequently, hydrogen, organic vapors, or acid vapors. Two methods of maintaining high purity in an inert atmosphere in a working enclosure are (1) continually flushing with a pure gas and (2) recycling the inert gas through a purification unit. As the volume of the enclosure and rate of contamination increase, economic considerations may favor the installation of a gas purification system. The problem then is to maintain the purity of the atmosphere in spite of continuing recontamination.

The solution:

Two systems which achieve and maintain a high level of gas purity have been developed for purifying inert atmospheres. One system, for helium, uses an activated charcoal bed at liquid nitrogen temperature to remove oxygen and nitrogen and uses molecular sieves to remove water vapor. The other system, for argon, uses heated (900°C) titanium sponge to remove nitrogen, copper wool beds to remove oxygen, and molecular sieves to remove water vapor. Impurity levels of <5 ppm each for O_2 and N_2 and <1 ppm H_2O vapor in the helium system and <20 ppm N_2, 5-15 ppm O_2, and 5-25 ppm H_2O vapor in the argon system are routinely obtained. These impurity levels are, however, dependent upon other variables such as enclosure volume, leak rate, diffusion rate through gloves, size and flow rate of the purification system, etc., but values such as these are considered typical for a well designed system.

How it's done:

In the helium purification unit shown in the left figure, the contaminated gas from the work enclosure first passes through a palladium catalyst. This unit reduces the hydrogen to 1 ppm by catalytic combination with oxygen to form water.

The molecular sieve bed absorbs water vapor to minimize ice or snow formation in the cold sections.

The heat exchanger, two concentric tubes formed into a single compact coil, is suspended in the vapor space above the boiling liquid nitrogen. The incoming

(continued overleaf)

gas flows in the annular section where it is cooled by the nitrogen vapor outside and the cold helium gas exciting from the inner tube.

The activated charcoal adsorber bed uses 8–14 mesh coconut charcoal in a copper vessel. This bed removes oxygen, nitrogen, and remaining traces of water vapor. To be most effective, the activated charcoal and the incoming gas stream should be at liquid nitrogen temperatures.

A "canned" blower returns the gas to the working enclosure. An eight-stage centrifugal blower, completely enclosed in a steel container, has operated satisfactorily for over 12,000 hr.

During dryer and adsorber regeneration, the boxes and blower are isolated. The carbon bed is regenerated by heating to ~150°C and then evacuating. The dryer is regenerated by heating to 300°C and passing 1 to 2 cfm of hot, dry nitrogen in reverse flow.

Operation of the purification unit at a total recirculation rate of ~10 cfm gave oxygen and nitrogen levels of <5 ppm and a moisture level of <1 ppm routinely to two 30-ft^3 volume work enclosures connected in parallel.

Argon System

Chemical "getters" such as Ti, Ca, V, and Ti–Zr alloy are generally used to remove nitrogen from argon. Titanium costs the least per mole of nitrogen removed and is readily available in sponge form. Oxygen is removed by passing the gas over a heated bed of copper wool.

In the argon purification system shown at the right a palladium catalyst bed removes the hydrogen as in the helium system. The water vapor also is adsorbed from the gas stream on a bed of molecular sieves. A filter removes any dust arising from the molecular sieve bed.

A critical component is the gas circulating pump which produces a discharge pressure sufficient to overcome the resistance of the titanium bed. Leak-tightness of the pump is required to avoid air and water-vapor contamination of the circulating gas. A rotary-vane pump is used to circulate the gas stream.

Remaining oxygen and water are removed by copper wool at 600°C. This step is particularly important because oxygen and water vapor passivate the titanium sponge with oxide, which inhibits the titanium–nitrogen reaction.

Nitrogen is removed by the titanium sponge at 900°C. For maximum conversion of the titanium sponge, the gas flow through the bed is periodically reversed to cause a more uniform conversion to titanium nitride.

The filter removes entrained particles. In operation, the circulation rate through the system is ~4 box-volumes/hr (2 cfm for 30-ft^3 enclosure).

Oxygen levels of 5 to 15 ppm and water levels of 5 to 25 ppm are routinely obtained. Low nitrogen levels in the working enclosure are more difficult to obtain because titanium is an effective getter for nitrogen only in the absence of oxygen and water vapor. In the absence of oxygen contamination in the inlet gas, nitrogen levels of <20 ppm can be maintained.

In both systems, the oxygen and moisture impurity levels are observed by electrolytic cells, in-line devices attached directly to the work enclosure. Nitrogen and hydrogen impurities are determined by standard chromatographic techniques. All methods have excellent accuracy in the 0 to 5 ppm range.

Notes:

1. The innovation is significant in that many chemical systems, i.e., metals, hygroscopic materials, and reactive materials, must be handled in a working enclosure.
2. Reference: Additional details may be found in *Nuclear Applications,* vol. 3, September 1967, p. 563–567.
3. Inquiries concerning this innovation may be directed to:

   Office of Industrial Cooperation
   Argonne National Laboratory
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   Reference: B69-10026

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Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

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